

A series of objectives of the new project S-1002, listed below, were emphasized by the participants in the first year of activity, 1 October 2001 – 30 September 2002, as described in the following:

**Objective 1. To Develop Value Added Products from Renewable/Recyclable Resources.**

*Synthesis and Characterization of Glucose-Based Polyhydroxylated Nylons (Glylons).* Glylon 6,6, which is the correspondent of petroleum-based Nylon 6,6, has been prepared at LSU-LA from activated glycaric acid (lactone form) and 1,6 hexamethylene diamine. Since the polymer decomposes right after melting (160-180 °C), a solvent has been identified for Glylons allowing the preparation of solutions for fiber spinning and film casting. Macroscopic spherulites (mm scale) have been formed in 2-20% concentrations. The higher the concentration, the smaller the spherulite dimensions. Composite blends have been prepared from Glylon 6, 6 and pulp (DP = 640). Concentrated solutions of Glylon 6, 6 and of blends showed a shear-induced organization which was shear-rate and temperature dependent. Large samples (up to 1/4 pound) have been prepared for advanced capillary elongation rheological characterization (ACER) and spinning of fiber through the cooperation with the University of Tennessee at Knoxville, TN.

*Characterization of plant-derived polyhydroxyalkanoates (PHAs).* A new LSU project concerned the characterization of plant-originating polyhydroxyalkanoates (PHAs), a family of aliphatic polyesters made by converting sunlight and carbon dioxide from the atmosphere using microbial or plant bio-factories. Work is underway in cooperation with Metabolix, Inc. from Cambridge, MA, the LSU Audubon Sugar Institute and the LSU Institute for Ecological Infrastructure Engineering to produce PHAs from cane sugar molasses by fermentation or directly from sugar cane plant - much like natural rubber is obtained directly from certain types of trees today. Preliminary data indicated that PHA obtained by fermentation has a melting temperature similar to that of polypropylene, making this polymer a candidate to be processed on the same equipment used for polyolefins. PHA melt blown nonwovens will be produced in cooperation with the TANDEC unit from the University of Tennessee in Knoxville, TN.

*Developing value-added products from bast fibers.* A preliminary investigation was directed at CO towards identifying natural colorants for dyeing of flax. Flax fabrics were dyed with four natural colorants of plant and insect origin. The colorants of plant origin were red sandalwood (*Pterocarpus santalinus*; active constituent: santalin), osage orange (*Maclura pomifera*; active constituent: tetrahydroxy stilbene compounds), madder root (*Rubia tinctorum*; active constituent: alizarin) and the colorant of insect origin was cochineal (*Dactylopius coccus*; active constituent: carminic acid). Pre-mordanting and simultaneous mordanting methods were compared. Tests were done to evaluate washing fastness, perspiration fastness and crocking fastness. The data illustrated that variation in origin and species, extraction procedure and dyeing procedure causes more variation with natural colorants than it would with synthetic dyestuffs of known compositions. The data also showed that the coloristic efficiency or tinctorial value of natural colorants on flax fabric was not very high, regardless of the order of mordanting. These low depths of

shade can be attributed to the high crystalline nature of flax fiber that results in slower diffusion and inferior dye penetration. Results of washing and perspiration fastness underscored the effect of pH on color of fabrics dyed with natural colorants. Crocking fastness was good for all the investigated natural colorants. It is hoped that with additional systematic research, natural colorants can be tailored to approach the stringent functional parameters of synthetic dyes and can be a viable commercial alternative for the coloration industry. Success in this area of research has potential in generating additional sources of income for U.S. agriculture. Initial studies are being conducted to determine the suitability of enzymes for wet processing of flax fiber. Cellulase, pectinase and viscozyme are some enzymes under study as potential biochemical replacements for traditional chemicals currently used by the industry.

*Use of Wood Fibers and Polymers for Preparation of Stable Sandwich-Type Materials.* Wood fibers were reacted at LSU with maleic anhydride in order to increase compatibility with synthetic polymers. Bread-butter-bread sandwich type laminates have been prepared from wood (bread) by hot-pressing using synthetic polymers (butter). Thermal transitions and mechanical properties were upgraded when maleic anhydride was added. Wood will be replaced with other fibrous cellulose derived from annual plants, such as bagasse and straw.

*Kenaf, Bagasse, and Ramie Fibers for Automotive Nonwovens.* Three types of nonwovens, kenaf/PP (70/30), bagasse/PP (50/50), and ramie/PP (70/30), were fabricated at LSU to construct two kinds of sandwich structures. The tailored nonwoven structures were suitable for molding in the manufacture of automotive interior trim parts. Tensile and compressive properties of the nonwoven structures were measured. Nonwoven thermal properties were characterized using the thermogravimetry, TG, and dynamo-mechanical analysis, DMA. Nonwoven absorbency after molding was also evaluated. Comparative analysis was carried out using the statistical methods of variance analysis and T-test. This work was done in collaboration with the USDA SRRC scientists.

*Chemical Treatment for Improving Kenaf Spinnability.* Kenaf fibers offer advantages of being renewable and environmentally safe. However, because raw kenaf fibers are usually short, coarse, and brittle, it is difficult to process them with conventional textile equipment. In this research, kenaf fiber bundles were treated by chemical methods and softened to improve fiber properties. Comparative analysis of the kenaf fiber in terms of fiber length, fiber fineness, and strength was done at LSU by Uster HVI, Uster Tensorapid, and Scanning Electron Microscopy (SEM) techniques. It was found that after the chemical treatment, fiber fineness, softness and elongation at break were improved, but the fiber bundle strength and length were decreased. Increasing the concentration of sodium hydroxide weaken the fiber strength significantly. Researches at LSU and SU-LA have continued collaborative research on improving kenaf yarns for apparel applications. Kenaf fiber was extracted from raw kenaf grown at Southern University or purchased from Mississippi State University using bacterial and chemical retting methods, then spun into kenaf/cotton yarns on ring or rotor spinning frames. Chemically retted kenaf was spun into yarn at SRRC.

*Textile Life Cycle Waste Management and Resource Recovery Model.* This is a new cooperative effort between University of Louisiana at Lafayette and LSU. Bread-butter-bread sandwich type nonwovens have been prepared based on bagasse and cotton webs (bread) using a solution of cellulose obtained from recyclable cotton fabrics (butter). Stable all cellulosic nonwoven materials have been obtained after pressing at 150°C, washing out the solvent and drying.

*To develop kenaf value-added products for textiles and crafts.* Raw kenaf plants, Everglades 41, were harvested from agronomic plot at Southern University and transported to LSU, SRRC in New Orleans, LA and AR for processing. AR performed biological retting method. The retted fibers were mailed to CO and SRRC for further processing and experiments. One value-added product, a tote bag, has been developed at AR using spun kenaf yarn. It has the capacity to stretch and hold at least ten Lbs. of goods.

*Development of Elastic and Absorbent Biodegradable Cotton-Surfaced Nonwovens (CSNs).* Cotton-rich webs (20-80%) were bonded at TN to one or both sides of a base structure, generally spunbond, SB, polypropylene, PP. Essentially biodegradable CSNs were produced by replacing the non-biodegradable PP SB substrate in the laminate with biodegradable polymer Eastar Bio GP Copolyester (Eastman Chemical Corporation). Furthermore, the thermoplastic PP staple fiber, which was blended with cotton fibers in carded or air-laid webs to improve thermal bonding of cotton-surfaced webs, was replaced with a largely biodegradable bi-component (bico) staple fiber with a core of PP (50%), to reduce elasticity for ease of carding, and a sheath of Eastar Bio (50%) for greater biodegradation and enhanced adhesion/thermal bonding. An essentially biodegradable CSN was prepared for ultrasonic bonding by laying an unbounded carded web of 70% bleached cotton and 30% bico staple fibers onto a SB polylactic acid, PLA provided by Cargill-Dow.

*Development of Absorbent Biodegradable Cotton-Core Nonwovens (CCNs).* These materials, developed at TN, are thermally bonded laminates having cotton cores with outer layers of melt blown, MB, or SB webs. In biodegradable CCNs PP in the SB and MB fabrics, as well as PP staple fibers in the cotton core, are replaced with biodegradable polymers. Physical test performed for CSNs and CCNs materials include determination of basis weight, air permeability, tenacity, tearing strength, wicking and water absorption properties.

## **Objective 2. To Develop Bioprocessing and Related New Technologies for Textiles.**

*Effect of Processing on Digitally Printed Fabrics.* Two main studies in digital textile printing were conducted at SU. The first investigated the effect of steaming time on color change and the second looked at colorfastness to crocking and laundering of digitally printed fabrics. Findings indicated that color change beyond one hour of steaming was not significant.

*One-Step Inkjet Printing and Durable Press Finishing of Cellulosic Fabrics.* University of Nebraska-Lincoln, Nebraska reported an inkjet printing technology that combines printing and durable press finishing in one process. Both acid and reactive dyes were examined. Alkaline catalysts were not required for reactive printing. Dimethyloldihydroxyethylene urea, DMDHEU, and butanetetracarboxylic acid, BTCA, were evaluated as crosslinking agents. Fabrics pretreated with crosslinkers and their catalysts and then printed with acid or reactive inks demonstrated that it was possible to inkjet print cotton fabrics with satisfactory dye fixation, colorfastness, and wrinkle resistant/DP properties. In most cases color yields of reactive dyes were better using this novel technology when compared to conventional alkaline fixation. The whole process was very similar to conventional inkjet printing (i.e., pretreatment, printing, steaming, drying and washing), with one additional step of curing (heat treatment at elevated temperature) after drying. It is a conventional step for textile mills, and also is readily achievable in a printing studio. Fabric pretreatment requires the conventional pad-dry process, which is exactly the same as normal pretreatment of inkjet fabrics. The only difference is that a crosslinker and its catalyst should be used instead of alkalis or some other chemicals. In addition to the improvement of wrinkle resistance and DP ability, this technology also provides possibilities of coloring cotton and other cellulosic fabrics with acid inks. Using a single set of inks for fabrics with different chemical structures could substantially decrease the waiting time and operating costs related to the ink changes in printing. It also provides a convenient solution to the printing of blends of cellulose with polyamides and or proteins using a single set of inks.

*Biodegradation of Effluents from Coloration of Textiles with Metallic Salts.* Biodegradation of all effluents from coloration of textiles (and leather) with metallic salts in conjunction with tannic acid occurred naturally in open atmosphere at ambient temperature. A series of microorganisms developed in waste waters from coloration of silk and leather, respectively, with gold, iron and titanium salts. They have been observed by optical microscopy and their identification is underway in order to “seed” the spent effluents with such organisms for a faster degradation.

### **Objective 3. To develop and evaluate textile systems for protective and medical applications.**

The potential for occupational exposure to blood borne pathogens, such as human immunodeficiency virus (HIV) and hepatitis B, has received much attention in recent years. Three major organizations have published guidelines for health care workers to minimize risks of exposure (Centers for Disease Control [CDC], Association of Operating Room Nurses [AORN], Occupational Safety and Health Administration [OSHA]). Use of protective apparel is a key factor in these recommendations. Protective surgical apparel can play an important role in minimizing disease transmission in the operating theater. Bacterial and viral diseases are spread through both airborne and blood borne pathways. Surgical apparel can reduce the transfer of microorganisms by creating

a physical barrier between the infection source and a healthy individual. Surgical gowns and medical facemasks are common protective apparel used by healthcare workers to reduce exposure. Fourteen medical face masks currently available on the market were selected for evaluation. Facemask characteristics of weight, thickness, pore size, repellency, moisture vapor transport, have been determined and the bacterial filtration efficiency (BFE) is currently being measured. Using BFE from 3 facemasks, a relationship between pore size and BFE was identified. As pore size decreases, BFE increases. The type of bacterial, *s. aureus* vs. *e. coli*, also influence BFE. Facemasks has higher BFE when *e. coli* was the challenging microorganism bacterial compared with *s. aureus*. The BFE was higher for *e. coli* as it is a rod shaped organism and *s. aureus* is round in shape. BFE of the remaining face mask is being determined. Fabrics representing common undergarment fabrics have been obtained and pre-washed and will be used in conjunction with surgical gown fabrics to determine if the type of undergarment fabrics has any impact on the barrier effectiveness of the surgical gown. Studies completed several years ago showed that there was a relationship between barrier effectiveness of outer garments of agricultural workers and the undergarment fabrics when evaluating pesticide transmission. Cotton and cotton polyester T-shirt fabrics have been obtained. Surgical gown fabrics are currently being selected. ASTM Test Method ASTM E 1670\_95 Standard Test Method for Resistance of Materials used in Protective Clothing to Penetration by Blood Borne Pathogens using Viral Penetration as a Test System is being modified to hold multiple fabric samples. Information gathered on the relationship between facemask characteristics and the Bacterial Filtration Efficiency will be used to engineer facemask with optimum filtration effectiveness. The impact of undergarments fabrics on the transmission of microorganisms and liquids through surgical gowns is of safety importance to healthcare workers.

#### **Objective 4. To Develop and Evaluate Textiles with Enhanced Resistance (or Susceptibility) to Environmental Degradation.**

*Preparation of Cotton/Bagasse/Kenaf Nonwoven for Horticulture End-Use.* This LSU research studied an approach to converting bagasse into a biodegradable nonwoven material for making flowerpots. A chemical method was used to extract bagasse fiber. The extracted fiber was cleaned and mixed with kenaf and cotton fibers with a ratio of 50:20:30. The fiber blend was carded and needle-punched to form nonwoven structure. The nonwoven fabric was further padded with starch paste and dried in an oven to increase nonwoven stiffness and strength. Application for horticulture container was studied. SEM analysis and tensile tester were used for evaluation of bonding structure and material strength decay after biodegradation.

*Preparation and characterization of nonwoven materials based on natural and synthetic fibers.* This LSU cooperation with TN, AR and the USDA SRRC allowed the preparation of biodegradable nonwoven composites based on bagasse, cotton, ramie, or kenaf fibers containing biodegradable polyester melt-bound nonwovens or lyocell derived from cotton solutions. Mechanical and thermal properties have been investigated and reported.

## **Publications:**

Chen, Y., Instrumental Method to Evaluate Leather Compressive Properties, Journal of Testing and Evaluation, 2002, 30, No.3, 258-261.

Chen, Y., O. Chiparus, X. Cui, T. Calamari and F. Screen, Bagasse Fiber Nonwoven Composite. Proceedings of 11<sup>th</sup> Annual International TANDEC Nonwovens Conference. November 6-8, 2001. The University of Tennessee, Knoxville, TN, pp. 3.6-1 – 3.6-10.

Chen, Y., O. Chiparus, X. Cui, T. Calamari and F. Screen, Cotton/Bagasse/Kenaf Nonwoven for Horticultural End-Use, Proceedings of Beltwide Cotton Conferences, January 2002, Atlanta, GA.

Chen, Y., O. Chiparus, I. Negulescu, D. V. Parikh and T. A. Calamari. Kenaf/Bagasse/Ramie Fibers for Automotive Nonwovens. Proceedings of 2002 International Nonwovens Technical Conference. Sept. 24-26, 2002, Atlanta, GA.

Chiparus, O., Negulescu, I., Chen Y. and Warnock, M., Nonwovens based on bagasse, kenaf and biodegradable polyesters, Book of Papers CD-ROM AATCC International Conference & Exhibition, Charlotte, North Carolina, October 1-4, 2002.

Collier, J. R., Negulescu, I. I. and Collier, B. J., US Patent 6,511,746 "Cellulosic Microfibers" issued to LSU on January 28 2003.

Cucu, M., Negulescu, I. I. and Laine, R. A., Polyhydroxylated Nylons: Glylon 6,6. Book of Papers CD-ROM AATCC International Conference & Exhibition, Charlotte, North Carolina, October 1-4, 2002

Denes, F., Manolache, S., Sarmadi, M., Ganapathy, R. and Martinez-Gomez, A., Process for Intercalation of Spacer Molecules Between Substrates and Active Biomolecules, US Patent 6,402,899, June 2002.

Huang, H.-Y., Characterization of Factors that Affect Charge Decay in Fibrous Electret, Ph. D. Dissertation, UT Knoxville, December 2001.

Kimmel, L., Negulescu, I., Chen, Y., Von Hoven, T., Graves, E. and Goynes, W., Naturally Colored Cotton for Specialty Textile Products, AATCC Review, Vol. 2(5), 25-29 (2002).

Leonas, K.K., Jones, C.R. and Shen, H. 2002. Medical Face Masks Fabrics: The Relationship of Permeability, Pore Size and Bacterial Transmission. INTC 2002 Proceedings of the International Nonwovens Technical Conference.

Lu, J. Z., Negulescu, I. I. and Wu, Q., Thermal and Dynamic-Mechanical Properties of Wood-PVC Composites, Book of Papers 6<sup>th</sup> Pacific Rim Bio-Based Composites Symposium, & Workshop on The Chemical Modification of Cellulosics, Portland, Oregon, November 10-13, 2002.

Lu, J. Z., Wu, Q. and Negulescu, I. I., The Influence of Maleation on Polymer Adsorption and Fixation, Wood Surface Wettability, and Interfacial Bonding Strength in Wood-PVC Composites, Wood and Fiber Science, 34(3), 434-459 (2002).

Ma, Y. C., Manolache, S., Sarmadi, M. and Denes, F., Plasma-Enhanced Maltodextrin-Polydimethylsiloxane Grafted Copolymers, J. Applied Polymer Science, 80(8), 1120-1128 (2001).

Ma, Y. C., Silicone Tetrachloride Plasma Induced Grafting for Starch-Based Composites, Ph.D. Dissertation, UW-Madison, August 2002.

McLean, E. C., Jr., Wadsworth, L. C., Sun, Q., Zhang, D. and Shaker, G., Development of Highly Absorbent Cotton-Core Nonwovens, Book of Papers CD-ROM INDA/TAPPI International Technical Conference, Atlanta, GA, September 24-26, 2002.

Negulescu, I., Chen, Y., Chiparus, O., Warnock, M. and Wadsworth, L., Biodegradable Nonwovens Based on Bagasse and Polyesters, Book of Papers CD-ROM 2002 Beltwide Cotton Conferences, Fifth Nonwovens Conference, Atlanta, GA, Jan 11-12, 2002.

Negulescu, I., Chen, Y., Chiparus, O., Warnock, M. and Wadsworth, L. and Yachmenev, V. G., Biodegradable Sandwich-Type Cellulosics/Polyester Nonwovens: Manufacture and Properties, Book of Papers CD-ROM INDA/TAPPI International Technical Conference, Atlanta, GA, September 24-26, 2002.

Negulescu, I., Young, N. and Todd, W., Metal Coloration of Textiles: Biodegradation of Residuals, Book of Papers CD-ROM AATCC International Conference & Exhibition, Charlotte, North Carolina, October 1-4, 2002

Parikh, D.V., Calamari, T. A., Sawhney, A. P. S., Briggs, R., Rigat, R. and Warnock, M. Improving Production Efficiency of a Cotton Swab Manufacturing Operation: A Case Study, Colourage, 35-42 (2002).

Parikh, D.V., Calamari, T. A., Sawhney, A. P. S., Blanchard, E. J., Screen, F. J., Warnock, M., Muller, D. H. and Stryjewski, D. D., Textile Research Journal, 72(7), 618-624 (2002).

Sarkar, A.K., Seal, C.M. and Willbur, J.S. (2002). Science and art of dyeing flax with natural dyes, International Textile and Apparel Association Annual Conference, New York, NY.

Schreuder-Gibson, H., Gibson, P. W., Wadsworth, L. C., Hemphill, S. M. and Vontorcik, J., Effect of Filter Deformation on the Filtration and Air Flow for Elastic Nonwoven Media, Proceedings, AFS 15<sup>th</sup> Annual Technical Conference and Exposition, American Filtration & Separation Society, Galveston, TX, April 9-12, 2002.

Shaker, G., A Study of Nonwoven Composites, M.S. Thesis, UT Knoxville, May 2002.

Sun, Q., Zhang, D., Wadsworth, L. C. and Slaten, B. L., Assessment of Comfort and Barrier Properties of Finished Cotton-Surfaced Nonwovens, Proceedings, Fourth International Nonwovens Symposium, Anaheim, CA, January 11-13, 2001, Session 6, Paper 3.

Wadsworth, L. C., Shaker, G., Zhang, D., Sun, Q. and McLean, E. C., Highly Absorbent Biodegradable Cotton Composites, Book of Papers CD-ROM 2002 Beltwide Cotton Conferences, Fifth Nonwovens Conference, Atlanta, GA, Jan 11-12, 2002.

Wadsworth, L. C., Sun, Q., Zhang, D., Zhao, R., Schreuder-Gibson, H. L. and Gibson, P., Process-Properties Study of Melt Blowing Polyurethane for Elastic Military Protective Apparel Garments, Book of Papers CD-ROM INDA/TAPPI International Technical Conference, Atlanta, GA, September 24-26, 2002.

Zhang, T., Y. Chen, G. Namwamba, D. Dixon, L. Kimmel and X. Cui. Chemical Treatment for Improving Kenaf Spinnability. Poster for 2002 AATCC International Conference and Exhibition. Oct. 1-4, 2002, Charlotte, NC.

Zhao, R., An Investigation of Bicomponent Polypropylene/Poly(Ethylene Terephthalate) Melt Blown Microfiber Nonwovens, Ph. D. Dissertation, UT Knoxville, December 2001.

Zhao, R., Wadsworth, L. C., Zhang, D. and Sun, C., Polymer Distribution During Bicomponent Melt Blowing of Polypropylene/Poly (Ethylene Terephthalate) and Its Improvement, J. Applied Polymer Science, 85, 2885-89 (2002).